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[MTD 13: Ores and Raw Materials]

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भाग 6 मुक्त सिलिका का निर्धारण

(पहला पुनरीक्षण)

Indian Standard

CHEMICAL ANALYSIS OF LIMESTONE, DOLOMITE AND ALLIED MATERIALS

PART 6 DETERMINATION OF FREE SILICA

(First Revision)

ICS 81.080

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Price Group 1

Methods of Chemical Analysis of Ores, Minerals and Allied Materials for Metallurgical Industry Sectional Committee, MTD 29

FOREWORD

This Indian Standard (Part 6) (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Ores, Minerals and Allied Materials for Metallurgical Industry Sectional Committee had been approved by the Metallurgical Engineering Division Council.

IS 1760 was first published in 1962. It covers the determination of different elements in various grades of minerals like limestone, dolomite, calcite and magnesite. It also covers the methods for magnesite refractories. The committee decided to revise this standard into different parts covering determination of each element in a separate part, which on publication would supersede the determination of that element given in IS 1760:1962. This Part covers determination of free silica by gravimetric method. The other parts in the series are as follows:

Part 1 Loss on ignition

Part 2 Determination of silica

Part 3 Determination of iron oxide, alumina, calcium oxide and magnesium oxide

Part 4 Determination of carbon dioxide

Part 5 Determination of chlorides

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical value (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

CHEMICAL ANALYSIS OF LIMESTONE, DOLOMITE AND ALLIED MATERIALS

PART 6 DETERMINATION OF FREE SILICA

(First Revision)

1 SCOPE

This standard (Part 6) describes the method for determination of free silica in limestone, dolomite and allied materials.

2 REFERENCES

The following standards contain provisions which through reference in this text, constitute provision of this standard. At the time of publication, the editions were valid. All standards are subject to revision , and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
2109 : 1982	Methods of sampling of limestone, dolomite and allied materials

3 SAMPLING

3.1 The sample shall be drawn and prepared in accordance with IS 2109.

3.2 Grind 5 to 10 g of the sample **3.1** so that it passes through IS sieve 150 microns opening. Dry to constant mass at 105 ± 2°C and use it for the purpose of chemical analysis.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and reagent grade water (*see* IS 1070) shall be employed for the test.

5 DETERMINATION OF FREE SILICA BY GRAVIMETRIC METHOD

5.1 Outline of the Method

After dissolution of a large sample of limestone or dolomite, in the acid, the insoluble matter including silica is separated, ignited at low temperature and residue left is fused with pyrosulphate. The silicic acid liberated from the clay minerals in the insoluble matter is dissolved in a hot solution of sodium hydroxide but the free silica is unaffected.

5.2 Reagents

5.2.1 *Dilute Hydrochloric Acid,* 1 : 1 (*v/v*).

5.2.2 *Potassium Bisulphate or Potassium Pyrosulphate*, solid.

5.2.3 *Sodium Hydroxide,* pellets.

5.2.4 *Dilute Sulphuric Acid,* 1 : 1 (*v/v*).

5.2.5 *Hydrofluoric Acid,* 40 percent (*v/v*).

5.3 Procedure

5.3.1 Weigh approximately 5.0 g (accurately weighed upto fourth decimal place) of the prepared sample in a 400 ml beaker, moisten it with little water and add 25 ml of dilute hydrochloric acid (1 : 1), and heat to boil. Filter the insoluble matter including silica and wash several times with hot water. Discard the filtrate.

5.3.2 Transfer the paper containing the insoluble matter in a platinum crucible and char the paper without inflaming at low heat. Increase the temperature to burn off the carbon, but do not exceed 600 ± 50°C. Cool and add approximately 3-5 g of fused and powdered potassium bisulphate or potassium pyrosulphate in the platinum crucible, and blend thoroughly with a small spatula. Fuse thoroughly over a gas burner, first by gradual heating to prevent loss of sulphur trioxide and then to a higher temperature with occasional stirring till the clear melt is obtained (*see* Note).

NOTE — Do not continue heating to a point where salts begin to freeze on top of the melt and on the sides of the crucible because of the difficulty of subsequent solution.

5.3.3 Cool the crucible and its contents and dissolve the melt by heating with 150 to 200 ml of water in a 400 ml beaker. To the warm solution cautiously and slowly with stirring add approximately 12 g of sodium hydroxide pellets, a few at a time to dissolve the precipitated silicic acid (*see* Note). Digest on a hot plate for 30 minutes at 80 to 90°C.

NOTE — Free silica is not transformed to silicic acid by fusion and is not affected by the caustic treatment.

5.3.4 Filter quickly, using a close-textured filter paper. Thoroughly scrub and wash the beaker with hot water. Wash the filter paper and its contents ten times with hot water, five times with hot dilute hydrochloric acid (1 : 1), to dissolve iron and other contaminants of the free silica and finally five times with hot water or until free of acid. Transfer the paper and residue to a tared platinum crucible. Ignite at 1 000°C to constant mass. Cool in a desiccator and weigh (*A*).

5.3.5 To the ignited and weighed residue (*A*) add 2 drops of dilute sulphuric acid (1 : 1) and approximately 10 ml of hydrofluoric acid. Evaporate to dryness, ignite and weigh (*B*) (*see* Notes 1 and 2).

NOTES

1 If the mass of the residue exceed 0.001 g,determination may have been improperly accomplished and hence discard and repeat the procedure specified in **5.3.1** to **5.3.4**.

2 Occasionally the free silica is contaminated by compounds not decomposed during fusion or dissolved in the subsequent treatment. The residue then is treated with hydrofluoric acid and sulphuric and silica expelled.

5.4 Calculation

Calculate the present free silica as follows:

$$\text{Free silica, percent by mass} = \frac{(A - B) \times 100}{m}$$

where

A = mass in g, of crucible and insoluble residue,

B = mass in g, of crucible after hydrofluorization ; and

m = mass in g, of the sample taken.

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